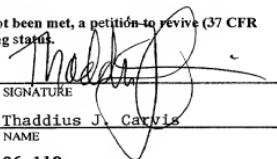


FORM PTO-1500 (REV. 11-2000)		U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE		ATTORNEY'S DOCKET NUMBER
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371				835-013.011-2
INTERNATIONAL APPLICATION NO. PCT/US00/25343		INTERNATIONAL FILING DATE September 15, 2000		U.S. APPLICATION NO. (If known, see 37 CFR 1.5 <b>09/831836</b>
TITLE OF INVENTION <b> SHEAR THICKENING PREGELATINIZED STARCH</b>				PRIORITY DATE CLAIMED September 15, 1999
APPLICANT(S) FOR DO/EO/US <b>Charles J. Brine, Anne E. Tielemans, Robert Wood</b>				
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:				
<p>1. <input checked="" type="checkbox"/> This is a <b>FIRST</b> submission of items concerning a filing under 35 U.S.C. 371.</p> <p>2. <input type="checkbox"/> This is a <b>SECOND</b> or <b>SUBSEQUENT</b> submission of items concerning a filing under 35 U.S.C. 371.</p> <p>3. <input checked="" type="checkbox"/> This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.</p> <p>4. <input checked="" type="checkbox"/> The US has been elected by the expiration of 19 months from the priority date (Article 31).</p> <p>5. <input type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371(c)(2))</p> <ol style="list-style-type: none"> <li><input type="checkbox"/> is attached hereto (required only if not communicated by the International Bureau).</li> <li><input type="checkbox"/> has been communicated by the International Bureau.</li> <li><input checked="" type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US).</li> </ol> <p>6. <input type="checkbox"/> An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).</p> <ol style="list-style-type: none"> <li><input type="checkbox"/> is attached hereto.</li> <li><input type="checkbox"/> has been previously submitted under 35 U.S.C. 154(d)(4).</li> </ol> <p>7. <input checked="" type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))</p> <ol style="list-style-type: none"> <li><input checked="" type="checkbox"/> are attached hereto (required only if not communicated by the International Bureau).</li> <li><input type="checkbox"/> have been communicated by the International Bureau.</li> <li><input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired.</li> <li><input type="checkbox"/> have not been made and will not be made.</li> </ol> <p>8. <input type="checkbox"/> An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).</p> <p>9. <input type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).</p> <p>10. <input type="checkbox"/> An English language translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).</p>				
<p><b>Items 11 to 20 below concern document(s) or information included:</b></p> <p>11. <input type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98.</p> <p>12. <input type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.</p> <p>13. <input checked="" type="checkbox"/> A <b>FIRST</b> preliminary amendment, with <b>four (4) sheets of new drawings</b>.</p> <p>14. <input type="checkbox"/> A <b>SECOND</b> or <b>SUBSEQUENT</b> preliminary amendment.</p> <p>15. <input type="checkbox"/> A substitute specification.</p> <p>16. <input type="checkbox"/> A change of power of attorney and/or address letter.</p> <p>17. <input type="checkbox"/> A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.</p> <p>18. <input type="checkbox"/> A second copy of the published international application under 35 U.S.C. 154(d)(4).</p> <p>19. <input type="checkbox"/> A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).</p> <p>20. <input type="checkbox"/> Other items or information:</p>				

U.S. APPLICATION NO. (if known) <u>09/831836</u>	INTERNATIONAL APPLICATION NO PCT/US00/25343	ATTORNEY'S DOCKET NUMBER 835-013.011-2		
<input checked="" type="checkbox"/> The following fees are submitted: <b>BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5):</b> Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO. .... <b>\$1000.00</b> International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO ..... <b>\$860.00</b> International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO ..... <b>\$710.00</b> International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) ..... <b>\$690.00</b> International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) ..... <b>\$100.00</b>		CALCULATIONS PTO USE ONLY		
<b>ENTER APPROPRIATE BASIC FEE AMOUNT</b> = <b>\$ 710.00</b> SurchARGE of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input checked="" type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(c)). <b>\$</b>				
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	\$
Total claims	25 - 20 =	5	x \$18.00	\$ 90.00
Independent claims	4 - 3 =	1	x \$80.00	\$ 80.00
<b>MULTIPLE DEPENDENT CLAIM(S) (if applicable)</b>			+ \$270.00	\$ 270.00
			<b>TOTAL OF ABOVE CALCULATIONS</b>	<b>\$1150.00</b>
<input checked="" type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2.			+ \$ 575.00	\$ 575.00
			<b>SUBTOTAL</b>	<b>\$ 575.00</b>
Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input checked="" type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(f)). <b>\$</b>				
			<b>TOTAL NATIONAL FEE</b>	<b>\$ 575.00</b>
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property + <b>\$</b>				
			<b>TOTAL FEES ENCLOSED</b>	<b>\$575.00</b>
			Amount to be refunded:	\$
			charged:	\$
a. <input checked="" type="checkbox"/> A check in the amount of <b>\$ 575.00</b> to cover the above fees is enclosed. b. <input type="checkbox"/> Please charge my Deposit Account No. _____ in the amount of \$ _____ to cover the above fees. A duplicate copy of this sheet is enclosed. c. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. <u>23-0442</u> . A duplicate copy of this sheet is enclosed. d. <input type="checkbox"/> Fees are to be charged to a credit card. <b>WARNING:</b> Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038.				
<b>NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137 (a) or (b)) must be filed and granted to restore the application to pending status.</b>				
SEND ALL CORRESPONDENCE TO: Thaddius J. Carvis Ware, Fressola, Van Der Sluys & Adolphson LLP Bradford Green, Building Five 755 Main Street, P.O. Box 224 Monroe, CT 06468 (203) 261-1234				
 SIGNATURE <u>Thaddius J. Carvis</u> NAME <u>26-110</u> REGISTRATION NUMBER				

09/831836  
JC18 Rec'd PCT/PTO 15 MAY 2001

**Practitioner's Docket No.** 835-013.011-2

## **PATENT**

**Preliminary Classification:**

**Proposed Class:**

**Subclass:**

NOTE: "All applicants are requested to include a preliminary classification on newly filed patent applications. The preliminary classification, preferably class and subclass designations, should be identified in the upper right-hand corner of the letter of transmittal accompanying the application papers, for example 'Proposed Class 2, subclass 129.' M.P.E.P. § 601, 7th ed.

**TRANSMITTAL LETTER TO THE U.S. DESIGNATED OFFICE (DO/US)—  
ENTRY INTO THE U.S. NATIONAL STAGE UNDER CHAPTER I**

INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/US00/25343	15 September 2000	15 September 1999

**TITLE OF INVENTION**

**SHEAR THICKENING PREGELATINIZED STARCH**

Box PCT  
Assistant Commissioner for Patents  
Washington, D.C. 20231

**ATTENTION: DO/US**

**CERTIFICATION UNDER 37 C.F.R. §§ 1.8(a) and 1.10\***

(When using Express Mail, the Express Mail label number is **mandatory**;  
Express Mail certification is **optional**.)

I hereby certify that, on the date shown below, this correspondence is being:

## MAILING

deposited with the United States Postal Service in an envelope addressed to the Assistant Commissioner for Patents, Washington, D.C. 20231  
37 C.F.R. § 1.8(a) 37 C.F.R. § 1.10 \*

with sufficient postage as first class mail.  as "Express Mail Post Office to Addressee"  
Mailbox Label No. EL176254038411S (mandatory)

## TRANSMISSION

facsimile transmitted to the Patent and Trademark Office, (703) \_\_\_\_\_ - \_\_\_\_\_

Date: 15 May 2001

Signature

Nancy S. Guth

(type or print name of person certifying)

**Only the date of filing (§ 1.6) will be the date used in a patent term adjustment calculation, although the date on any certificate of mailing or transmission under § 1.8 continues to be taken into account in determining timeliness. See § 1.703(d). Consider "Express Mail Post Office to Addressee" (§ 1.10) or facsimile transmission (§ 1.5(d)) for the reply to be accorded the earliest possible filing date for patent term adjustment calculations.**

(Transmittal Letter to the United States Designated Office (DO/US)—Entry into National Stage under  
35 U.S.C. § 371 [13-6]—page 1 of 9)

(835-013.011-2)

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re Application of :  
CHARLES J. BRINE, et al :  
International application No. PCT/US00/25343 :  
Filed September 15, 2000 :  
For: SHEAR THICKENING PREGELATINIZED :  
STARCH :  
:

BOX PCT  
Assistant Commissioner for Patents  
Washington, D. C. 20231  
**Attention: DO/US**

Sir:

Kindly enter the following Preliminary Amendment and Remarks in this application after grant of a serial number, but prior to examination.

**PRELIMINARY AMENDMENT**

In the Specification:

On page 2, replace the last two paragraphs with the following paragraphs:

Figure 1 is a graph which compares the effect shear has on a model food system employing a preferred starch according to the invention (-♦-) with two that are not, namely a granular CWS medium cross linked potato starch (-■-) and a nongranular CWS high cross linked potato starch (-●-).

**Express Mail Certificate:** I hereby certify that this correspondence is today being deposited with the U.S. Postal Service as "Express Mail Post Office to Addressee" Mailing Label Number EL762540384US in an envelope addressed to: Assistant Commissioner of Patents, BOX , Washington, D.C. 20231.

May 15, 2001

*Nancy S. Guth*  
Nancy S. Guth

Figure 2 is a chart showing the Brabender viscosity characterizations of a source potato starch used to produce a preferred starch of the invention (— • —) in comparison to other source starches, namely a medium cross-linked potato starch (— —) and low cross-linked potato starch (• • •), prior to the atomized (spray) cooking process. The temperature variation with time is also shown (—).

On page 3, replace paragraphs 1 and 2 with the following paragraphs:

Figure 3 is a graph which shows the effect of shear on viscosity on salad dressings prepared with a preferred starch of the invention (-♦-) and two other starches, namely a granular CWS medium cross linked potato starch (-■-) and a nongranular CWS high cross linked potato starch (-●-).

Figure 4 is a graph illustrating one difference, namely lack of viscosity build-up under low shear conditions, between a preferred starch of the invention (lower curve) and a CWS highly-crosslinked waxy maize starch (upper curve).

On Page 8 replace paragraphs 3 and 4 with the following paragraphs:

Figure 1 illustrates the above significant characterizing properties of the starch of the invention. Figure 1 is a graph which compares the effect shear has on a model food system employing a preferred starch according to the invention (-♦-) with two that are not, namely a granular CWS medium cross linked potato starch (-■-) and a nongranular CWS high cross linked potato starch (-●-). The data for the figure was obtained for an aqueous food system such as what may be found in a pourable, fat-free salad dressing. The food system was manufactured using three different starch preparations as described in Example 1. Any cold water swelling starch that exhibits a similar increase in viscosity with high shear, e.g., not less than 50% of the values of the preferred starch illustrated in Figure 1 (-♦-) is within the scope of this invention. The viscosity after high shear should be double or preferably 6-7 times as high as that exhibited after low shear and should not decrease in viscosity from a maximum by more than about 25%.

Figure 2 is a chart showing the Brabender viscosity characterizations of a source potato starch used to produce a preferred starch of the invention (— • —) in

comparison to other source starches, namely a medium cross-linked potato starch (— —) and low cross-linked potato starch (• • •), prior to the atomized (spray) cooking process. The temperature variation with time is also shown (—). Preferred is a starch that has a Brabender curve similar to that of the high crosslinked starch with a peak viscosity less than 600 Bradender Units. The most preferred starch is potato but could be, especially, other non-cereal starches (e.g. , tapioca or arrow root).

On page 9 replace the first two paragraphs with the following:

Figure 3 is a graph which shows the effect of shear on viscosity on salad dressings prepared with a preferred starch of the invention (-♦-) and two other starches, namely a granular CWS medium cross linked potato starch (-■-) and a nongranular CWS high cross linked potato starch (-●-). It can be seen that when a modified potato starch such as the preferred starch is employed in a model formulation such as in Example 1 where high shear is intrinsic to the model salad dressing production, the after high shear viscosity of the model dressing is 7-8 times as high as that observed after low shear mixing. Other salad dressing starches such as similarly modified waxy maize, and similarly modified corn starch, do not show such increases in viscosity with high shear when processed under the conditions employed for the potato starch.

Figure 4 is a graph illustrating one difference, namely lack of viscosity build-up, between a preferred starch of the invention, namely a CWS highly-crosslinked potato starch (lower curve), and a similar CWS highly-crosslinked waxy maize starch (upper curve) under low shear conditions. The graph was prepared by dispersing in separate vessels, 1.4 g each of the starches with 2.8 grams of glycerine and 23.8 grams of water. The samples were then tested using the Instruction Manual procedures (March 1998) for Rapid Visco Analyser series 4 manufactured by Newport Scientific Pty. Ltd., Australia. The following Profile settings were employed: Temperature- 25°C; Initial rpm - 960 rpm for 10 seconds; Run rpm - 60 rpm for 15 minutes. As noted above, the preferred starches are characterized in that slurries of them do not significantly increase viscosity under low shear conditions, the increase being less than 50%, and preferably less than 25%, of the maximum achievable under high shear conditions.

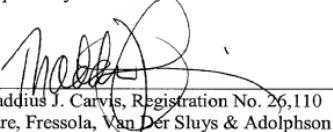
In the Drawings:

Replace the four (4) sheets of drawings originally filed in International Application PCT/US00/25343 with the enclosed four (4) sheets of new drawings.

**REMARKS**

Entry of the above amendments is in order and such action is earnestly solicited. These amendments were previously made in the international application in response to an invitation to correct informalities.

Respectfully submitted,



Thaddius J. Carvis, Registration No. 26,110  
Ware, Fressola, Van Der Sluys & Adolphson LLP  
Bradford Green, Building Five  
755 Main Street, P.O. Box 224  
Monroe, CT 06468  
(203) 261-1234

Customer No. 004955

## MARKED UP PARAGRAPHS

### Page 2, last two paragraphs:

Figure 1 is a graph [titled “Model Food System Viscosity Profile”] which compares the effect shear has on a model food system employing a preferred starch according to the invention (-♦-) with two that are not, namely a granular CWS medium cross linked potato starch (-■-) and a nongranular CWS high cross linked potato starch (-●-).

Figure 2 is a chart showing the Brabender viscosity characterizations of a source potato starch used to produce a preferred starch of the invention (— • —) in comparison to other source starches, namely a [medium cross-linked potato starch (— — —) and low cross-linked potato starch ()] (● • ●), prior to the atomized (spray) cooking process. The temperature variation with time is also shown (—).

### Page 3, Paragraph 1

Figure 3 is a graph [titled “Basic Salad Dressing Viscosity Profile”] which shows the effect of shear on viscosity on salad dressings prepared with a preferred starch of the invention (-♦-) and two other starches, namely a granular CWS medium cross linked potato starch (-■-) and a nongranular CWS high cross linked potato starch (-●-).

### Page 3, Paragraph 2

Figure 4 is a graph illustrating one difference, namely lack of viscosity build-up under low shear conditions, between a preferred starch of the invention (lower curve) and a CWS highly-crosslinked waxy maize starch (upper curve).

### Page 8, Paragraph 3

Figure 1 illustrates the above significant characterizing properties of the starch of the invention. Figure 1 is a graph [titled “Model Food System Viscosity Profile”] which compares the effect shear has on a model food system employing a preferred starch according to the invention (-♦-) with two that are not, namely a granular CWS medium cross linked potato starch (-■-) and a nongranular CWS high cross linked potato starch (-●-). The data for the figure was obtained for an aqueous food system such as what may be found in a pourable, fat-free salad dressing. The food system was manufactured using

three different starch preparations as described in Example 1. Any cold water swelling starch that exhibits a similar increase in viscosity with high shear, *e.g.*, not less than 50% of the values of the preferred starch illustrated in Figure 1 (-♦-) is within the scope of this invention. The viscosity after high shear should be double or preferably 6-7 times as high as that exhibited after low shear and should not decrease in viscosity from a maximum by more than about 25%.

Page 8, Paragraph 4

Figure 2 is a chart showing the Brabender viscosity characterizations of a source potato starch used to produce a preferred starch of the invention (— • • —) in comparison to other source starches, namely a [□] medium cross-linked potato starch (—  
— — —) and low cross-linked potato starch [○] (● ● ●), prior to the atomized (spray) cooking process. The temperature variation with time is also shown (—). Preferred is a starch that has a Brabender curve similar to that of the high crosslinked starch with a peak viscosity less than 600 Bradender Units. The most preferred starch is potato but could be, especially, other non-cereal starches (*e.g.* , tapioca or arrow root).

Page 9, Paragraph 1

Figure 3 is a graph [titled "Basic Salad Dressing Viscosity Profile"] which [describes a practical application of this invention.] shows the effect of shear on viscosity on salad dressings prepared with a preferred starch of the invention (-♦-) and two other starches, namely a granular CWS medium cross linked potato starch (-■-) and a nongranular CWS high cross linked potato starch (-●-). It can be seen that when a modified potato starch such as the preferred starch is employed in a model formulation such as in Example 1 where high shear is intrinsic to the model salad dressing production, the after high shear viscosity of the model dressing is 7-8 times as high as that observed after low shear mixing. Other salad dressing starches such as similarly modified waxy maize, and similarly modified corn starch, do not show such increases in viscosity with high shear when processed under the conditions employed for the potato starch.

Page 9, Paragraph 2

Figure 4 is a graph illustrating one difference, namely lack of viscosity build-up, between a preferred starch of the invention, namely a CWS highly-crosslinked potato

starch (lower curve), and a similar CWS highly-crosslinked waxy maize starch (upper curve) under low shear conditions. The graph was prepared by dispersing in separate vessels, 1.4 g each of the starches with 2.8 grams of glycerine and 23.8 grams of water. The samples were then tested using the Instruction Manual procedures (March 1998) for Rapid Visco Analyser series 4 manufactured by Newport Scientific Pty. Ltd., Australia. The following Profile settings were employed: Temperature- 25°C; Initial rpm - 960 rpm for 10 seconds; Run rpm - 60 rpm for 15 minutes. As noted above, the preferred starches are characterized in that slurries of them do not significantly increase viscosity under low shear conditions, the increase being less than 50%, and preferably less than 25%, of the maximum achievable under high shear conditions.

4 pts

WO 01/19404

09/831836  
10 REG. NO. 15 MAY 2001  
PCT/US00/25343

## DESCRIPTION

5

### Shear Thickening Pregelatinized Starch

#### Background of The Invention

10 The invention relates to an improved starch product, products employing it and to processes for making and using it. In particular, it relates to shear thickening pregelatinized starch and its use in the preparation of a variety of products including foods and cosmetics.

15 It is known that starch becomes functional for thickening and texturizing and/or rheologically altering various products, including but not limited to foods, only after being gelatinized or cooked in aqueous slurry beyond a critical temperature (*i.e.*, the gelatinization point) such that the granules swell, absorb water and increase the slurry viscosity. For convenience, starch manufacturers offer dry starches which have already been cooked for the user, so-called pregelatinized starches. This pregelatinization has commonly been carried out by jet cooking, roll drum drying, extrusion cooking and recently by atomized or spray cooking processes.

20 These methods produce pregelatinized starches of a type known as cold water swelling "CWS" starches, which exhibit a range of granularity (*i.e.*, fully ruptured to totally intact granules). Cold water swelling starches are distinguished from cold water soluble starches 25 because the former retains its granularity while the latter is fully soluble and largely non-granular. It has been accepted or shown by those skilled in the art that further processing with heat, acid and/or shear, as typical in food processing, would tend to reduce the peak viscosities of the pregelatinized starches relative to those initially observed for each prior to such processing stress (see, for example, D. J. Thomas and W. A. Atwell, *Starches*, 1999). This 30 observed relative viscosity reduction versus shear has been considered to be independent of the raw material source of the starch. While chemical modifications (*e.g.*, esterification, etherification, phosphorylation, hydrolysis, oxidation and/or crosslinking) made to alter starch functionality can mitigate viscosity reductions from shear, they do not eliminate them entirely (Whistler, et al, *Starch: Chemistry and Technology*, 1984). Clearly, increases in viscosity with

shear of the magnitude described by this invention previously have not been attributed to such modifications. Indeed, the art has been without a starch having this unique set of characteristics.

5 There is a need in the art for pregelatinized starches which are relatively stable (freeze/thaw, refrigerated storage, heat, shear), yet can be easily further processed at low, workable viscosities. There is a need for such starches that build higher, desirable product viscosities upon application of significant mechanical action (e.g., shear by various mixers, Lightnin™ Mixer, Waring™ blender, colloid mill, etc.). In several industries including food processing, cosmetics, personal care, among others, such characteristics in a thickening or rheological ingredient would be advantageous and desirable.

10 This novel combination of properties for pregelatinized starches has not been reported previously and will have useful applications in numerous commercial areas (e.g., food processing, cosmetics, personal care, etc.). Food applications where this thin-to-thick ("thin-thick") rheological profile would be useful include, but are not limited to, pourable salad dressings, spoonable mayonnaise and dressings and fruit preparations. Cosmetic and personal care applications include, but are not limited to, skin and body lotions, hair creams and preparations and cosmetic creams and lotions.

20

#### **Brief Description of the Drawings**

25 The invention will be better understood and its advantages more apparent from the following description, especially when read in light of the accompanying drawings, wherein:

Figure 1 is a graph titled "Model Food System Viscosity Profile" which compares the effect shear has on a model food system employing a preferred starch according to the invention with two that are not.

30 Figure 2 is a chart showing the Brabender viscosity characterizations of a source potato starch used to produce a preferred starch of the invention in comparison to other source starches, (medium cross-linked potato starch and low cross-linked potato starch), prior to the atomized (spray) cooking process.

Figure 3 is a graph titled "Basic Salad Dressing Viscosity Profile" which shows the effect of shear on viscosity on salad dressings prepared with a preferred starch of the invention and two other starches.

Figure 4 is a graph illustrating one difference, namely lack of viscosity build-up under 5 low shear conditions, between a preferred starch of the invention and a CWS highly-crosslinked waxy maize starch.

### Summary of the Invention

10

It is an object of the invention to provide a new pregelatinized, cold water swelling starch, a slurry of which exhibits a large increase in viscosity to a maximum following high shear mixing.

15

It is an object of the invention to provide a new pregelatinized, cold water swelling starch, a slurry of which exhibits a large increase in viscosity to a maximum following high shear mixing but only a modest increase following low shear mixing.

20

It is another object of the invention to provide a new pregelatinized, cold water swelling starch, a slurry of which exhibits an increase in viscosity to a maximum following high shear mixing wherein the final viscosity following mixing and holding to form a product is no less than 25% of the maximum viscosity.

25

It is another and more specific object of the invention to provide a new pregelatinized, cold water swelling starch, a slurry of which exhibits an increase in viscosity to a maximum following high shear mixing and which are relatively stable (freeze/thaw, refrigerated storage, heat, shear).

30

It is yet another and more specific object of the invention to provide a new pregelatinized, cold water swelling starch, a slurry of which can be easily processed at low, workable viscosities under low shear conditions without significant viscosity increased, yet exhibits an increase in viscosity to a maximum following high shear mixing.

It is yet another and more specific object of the invention to provide a new pregelatinized, cold water swelling starch, a slurry of which exhibits an increase in viscosity to a maximum following high shear mixing and thereby build higher, desirable product viscosities upon application of significant mechanical action (e.g., shear by various mixers, 5 Lightnin™ Mixer, Waring™ blender, colloid mill, etc.) in preparing products in several industries including food processing, cosmetics, personal care among others where such characteristics in a thickening or rheological ingredient are advantageous and desirable.

10 It is yet another and more specific object of the invention to provide a new pregelatinized, cold water swelling starch, a slurry of which exhibits an increase in viscosity due to high shear mixing and can beneficially improve a wide variety of products, including food, cosmetics and other consumer and industrial products that involve high shear mixing during preparation or prior to use.

15 These and other objects are accomplished by the invention, which provides a process for a new pregelatinized, cold water swelling starch, comprising: providing a highly cross linked, stabilized starch; and heating said starch under conditions of time, temperature and moisture effective to disrupt less than 50% of intact starch granules comprised in said starch 20 and to provide the characteristic that when a slurry of said starch is subjected to high shear the viscosity will increase toward a maximum and retain this viscosity throughout further low shear mixing and holding periods.

In another aspect, the invention provides a pregelatinized starch product, which 25 exhibits shear-thickening properties, comprising: a highly crosslinked, stabilized and cold water swelling starch having a majority of its starch granules intact and being characterized in that when a slurry of said starch is subjected to moderate to high shear the viscosity will increase toward a maximum and retain that viscosity throughout further low shear mixing and holding periods.

Among the preferred starches are root or tuber starches, particularly potato starch, including those derived from white potatoes, yams and sweet potatoes, among others.

5 The process has a number of preferred aspects, which are described below and shown in the attached drawings.

### **Detailed Description of The Invention**

This description will describe a unique new starch product and processes for preparing and using it in terms of the preferred mode, which is based on potato starch. Those skilled in the art will recognize that some, if not all, of the advantages of the invention can be achieved for starches from other sources. Similarly, the product applications described are presently preferred, but only exemplary. The invention has wide application to a variety of products, including, but not limited to, foodstuffs such as salad dressings or mixes thereof, cosmetics and personal care products. These products and others can be improved due to the presence of these new properties discovered for a category of gelatinized starch products which are the objects of this invention.

10 Those skilled in the art know that starch is deposited in various plants in the form of minute cold water insoluble granules. The sources of starches include seeds of plants such as wheat, sorghum and rice, tubers such as potato (including those derived from white potatoes, yams and sweet potatoes, among others), cassava and arrowroot and the pith of plants such as the sago palm and other sources including tapioca and the like. The source of the starch and its processing history will cause its properties to vary. The nature of the starch granules and 15 the makeup of the starch can vary among the starches from the various sources. The work underlying the invention has been conducted principally on potato starch, but there is no reason known to exclude starches from any particular source. Thus, other starches including other root starches such as arrowroot starch and tapioca starch are included as source materials.

The skilled worker will vary the processing specific for potato to achieve success for other starches. The success in achieving the invention, regardless of source of starch will be determined on the basis of whether a starch meets the criteria of the invention which are: cold water solubility; with a viscosity profile that increases to a maximum with the application of 5 moderate to high shear to a slurry of the starch and maintains that viscosity upon storage or under low shear for at least one week, preferably at least one month thereafter. All successful products will be highly crosslinked and have at least 50% of the starch granules intact in addition to being cold water swelling. However, not all starches meeting these last criteria will have the unique characteristic of a viscosity profile that increases to a maximum with the 10 application of moderate to high shear to a slurry of the starch and maintains that viscosity upon storage or under low shear. Also, the preferred starches are characterized in that slurries of them do not significantly increase viscosity under low shear conditions, the increase being less than 50%, and preferably less than 25%, of the maximum achievable under high shear conditions.

15

As used herein, the term "cold water swelling" refers to a pregelatinized starch that maintains its granular shape when rehydrated. And, the term "cold water soluble" refers to a pregelatinized starch with little or no granular integrity. The amylose and amylopectin are solubilized upon dehydration.

20

For the purposes of this description, the term "granularity" means the amount of starch present as intact granules as determined by microscopic examination of a known concentration of starch in aqueous dispersion. And, the term "Granular" refers to starch present as intact granules, whether pregelatinized or uncooked.

25

Because the amount of shear produced by various equipment will vary widely it is necessary to provide an objective meaning for the terms "low shear" and "high shear" as used herein. Thus, we define the terms low and high using several pieces of conveniently-available laboratory equipment, namely a Caframo-Heidolph, Model # RZR 1, Stirrer and a Waring<sup>TM</sup> 30 Commercial Laboratory Blender, Model 34BL97 By Waring Co., New Hartford, CT.

"High shear" is defined using a Waring Blender and the following procedure.

### High Shear Measurement – Brookfield viscosity method

Formulation – 750g

5	a. 5% Starch	37.5g
	b. 5% Sugar	37.5g
	c. Deionized Water	337.5g
	d. Vinegar (3%)	337.5g

10      **Equipment**

Blender: Waring Commercial Laboratory Blender, Model #34BL97

15      **Procedure**

- a. Measure out the ingredients as listed above.
- b. Combine the dry starch and sugar by placing both into a bag or bottle mixing thoroughly by shaking vigorously for 1 minute.
- c. Combine water and vinegar in a 40-oz. blender vessel at lowest speed(setting #1).
- d. Add the dry mix slowly to the blending water/vinegar solution until all the mixture has dispersed.
- e. Increase blender speed to maximum (setting #7) and mix for 30 seconds.
- f. Stop blender and pour suspension into a 600ml beaker. Allow starch suspension to rest quiescently.
- g. Set up a Brookfield RTV viscometer with spindle #4 set at 10rpm.
- h. After 15 minute of quiescent resting, immerse the spindle in the starch suspension and run for 1 minute. Measure the Brookfield viscosity using spindle #4, 10rpm.

20      "Low shear" is defined using a Caframo-Heidolph, Model # RZR 1, Overhead Mixer and the following procedure.

25      **Low Shear Measurement – Brookfield viscosity method**

Formulation – 750g

30	a. 5% Starch	37.5g
	b. 5% Sugar	37.5g
	c. Deionized Water	337.5g
35	d. Vinegar (3%)	337.5g

40      **Equipment**

Stirrer: Caframo-Heidolph, Model # RZR 1  
w/3-blade impeller

## Procedure

- a. Measure out the ingredients as listed above.
- b. Combine the dry starch and sugar by placing both into a bag or bottle mixing thoroughly by shaking vigorously for 1 minute.
- 5 c. Add the dry mix slowly to the moderately stirring(forms vortex, stirrer setting #2) water/vinegar solution until all the mixture has dispersed. Then increase stirring speed to form high vortex (stirrer setting #4) for 60 seconds.
- d. Stop stirring and allow starch suspension to rest quiescently.
- 10 e. Set up a Brookfield RTV viscometer with spindle #4 set at 10rpm.
- f. After 15 minutes of quiescent resting, immerse the spindle in the starch suspension and run for 1 minute. Measure the Brookfield viscosity using spindle #4, 10rpm.

Thus, regardless of the mixing one actually uses in production or processing when employing the starches of the invention, the above tests are used to determine the degree of shear for which a viscosity change will be observed. In other words, we use this test to provide definition to the terms in this description and the claims and this test will then be used to determine if a starch product meets the terms of the claims.

Figure 1 illustrates the above significant characterizing properties of the starch of the invention. Figure 1 is a graph titled "Model Food System Viscosity Profile". The data for the figure was obtained for an aqueous food system such as what may be found in a pourable, fat-free salad dressing. The food system was manufactured using three different starch preparations as described in Example 1. Any cold water swelling starch that exhibits a similar increase in viscosity with high shear, e.g., not less than 50% of the values of the preferred starch illustrated in Figure 1 is within the scope of this invention. The viscosity after high shear should be double or preferably 6-7 times as high as that exhibited after low shear and should not decrease in viscosity from a maximum by more than about 25%.

Figure 2 is a chart showing the Brabender viscosity characterizations of the source potato starch used to produce the preferred starch in comparison to other source starches, (medium cross-linked potato starch and low cross-linked potato starch), prior to the atomized (spray) cooking process. Preferred is a starch that has a Brabender curve similar to that of the high crosslinked starch with a peak viscosity less than 600 Brabender Units. The most preferred starch is potato but could be, especially, other non-cereal starches (e.g., tapioca or arrow root).

Figure 3 is a graph titled "Basic Salad Dressing Viscosity Profile" which describes a practical application of this invention. It can be seen that when a modified potato starch such as the preferred starch is employed in a model formulation such as in Example 1 where high shear is intrinsic to the model salad dressing production, the after high shear viscosity of the model dressing is 7-8 times as high as that observed after low shear mixing. Other salad dressing starches such as similarly modified waxy maize, and similarly modified corn starch, do not show such increases in viscosity with high shear when processed under the conditions employed for the potato starch.

10

15

20

Figure 4 is a graph illustrating one difference, namely lack of viscosity build-up, between a preferred starch of the invention, namely a CWS highly-crosslinked potato starch, and a CWS highly-crosslinked waxy maize starch under low shear conditions. The graph was prepared by dispersing in separate vessels, 1.4 g each of the starches with 2.8 grams of glycerine and 23.8 grams of water. The samples were then tested using the Instruction Manual procedures (March 1998) for Rapid Visco Analyser series 4 manufactured by Newport Scientific Pty. Ltd., Australia. The following Profile settings were employed: Temperature- 25°C; Initial rpm - 960 rpm for 10 seconds; Run rpm - 60 rpm for 15 minutes. As noted above, the preferred starches are characterized in that slurries of them do not significantly increase viscosity under low shear conditions, the increase being less than 50%, and preferably less than 25%, of the maximum achievable under high shear conditions.

25

The starch products referred to in all the figures, tables and examples below are defined and characterized as per Table 1. Crosslinking was carried out for all using phosphorous oxychloride ( $\text{POCl}_3$ ) following standard procedures. Stabilization, hydroxypropylation (HP), was carried out for all with propylene oxide following standard procedures.

**Table 1. Identification and characterization of starch products referred to in figures, tables and examples**

Starch	Source	Cooking Method	Crosslinking $\text{POCl}_3$ (ppm)	Substitution	DS
Granular CWS high cross-linked potato starch*	PS	AC	75-400	HP	0.8-0.16
Granular CWS medium cross-linked potato starch	PS	AC	45-74	HP	0.8-0.16
Non-Granular CWS high cross-linked potato starch	PS	DD	75-400	HP	0.8-0.16
Granular CWS low cross-linked potato starch	PS	AC	15-44	HP	0.8-0.16
Granular CWS high cross-linked waxy maize starch	WM	AC	75-400	HP	0.8-0.16
Granular CWS high cross-linked corn starch	C	AC	75-400	HP	0.8-0.16
Granular CWS medium cross-linked waxy maize starch	WM	AC	45-74	HP	0.8-0.16

5 PS = Potato Starch

WM = Waxy Maize C = Corn Starch

AC = Atomized (Spray) Cooking DD = Roll Drum Dried

DS = degree of substitution as determined by test method described in The United States Pharmacopoeia (USP 24) pp. 843-844. The same test is used for cellulose as for starch.

10 The assay is also known as the Zeisel Method.

"Granular CWS high cross-linked potato starch" is the preferred starch based on testing to date.

The invention achieves the above properties for a cold water swelling starch by

15 obtaining a highly crosslinked, stabilized starch and heating this starch under conditions of time, temperature and moisture effective to disrupt less than 50% of intact starch granules comprised in said starch and to provide the characteristics described herein. A preferred

process for heating is described in a series of U.S. patents (U.S. Patent No. 4,280,851 issued July 28, 1981 and U.S. Patent No. 4,600,472 issued July 15, 1986 to Pitchon, *et al.*, U.S. Patent No. 4,847,371 issued July 11, 1989 to Schara, *et al.*). These patents teach a method for atomized or spray cooking of starch, the process involved, the equipment design, *etc.* in 5 operating the process of Pitchon, *et al.*, the conditions of operation will be selected to achieve the objective product criteria set out herein. The specific equipment configuration will to some extent require trial and error to arrive at the optimum conditions in terms of achieving the properties of shear-related viscosity and granularity required. In addition to these methods, other heating methods such as drum drying, jet cooking, extrusion cooking and the like can 10 be employed when, again, operated under conditions effective to achieve the objective product criteria set out herein.

15  

As used herein, the term "crosslinked starch" refers to modified starches in which chemical bonds (bridges) in and between starch molecules have been formed by means of bifunctional or polyfunctional chemical reagents. Crosslinked starches are made by treating starch in the granular state with these reagents which are capable of reacting with the hydroxyl groups in and between the starch molecules or molecule chains within the granule. The amount of crosslinking reagent is generally small since only one crosslink is required for every 100 to 2000 starch units to achieve the desired functional effect. The degree of crosslinking 20 controls the rate and extent of starch granule swelling during cooking. Functionally, crosslinking is employed to impart greater heat resistance, acid stability and shear tolerance to the starch. In general, reagents such as phosphorus oxychloride, sodium trimetaphosphate and adipic acid may be used as crosslinking agents. In the preferred embodiment all crosslinking was carried out using phosphorus oxychloride (POCl<sub>3</sub>) as per the conditions 25 listed in Table 1.

As used herein, the term "stabilized starch" refers to are modified starches which have been protected against retrogradation by using monofunctional reagents which chemically react with the hydroxyl groups on the starch molecules to introduce a substituent groups. These 30 substituent groups interfere with or sterically hinder the intermolecular association between dissolved amylose molecules in starch pastes, slurries and solutions. Functionally, stabilization is employed to reduce gelatinization temperature, improve water uptake and

retention and improve freeze/thaw stability. Examples of stabilized food starches are monostarch phosphates, acetylated starches and hydroxypropylated starches. Generally, the degree of substitution (DS) of these starches is between 0.01 and 0.2 (*i.e.*, 1 to 20 substituent groups introduced per 100 starch units). In the preferred embodiment all stabilization was 5 carried out using propylene oxide as per the conditions in Table 1 to effect hydroxypropylation (HP).

In a preferred embodiment, the invention provides for the production of a starch, Granular CWS high cross-linked potato starch in Table 1, by practicing the atomized cooking 10 technology detailed in U.S. Patent Nos. 4,280,851, 4,600,472 and 4,847,371 whereby 30-40% solids starch slurries at a pH = 6.0 – 7.5 are pumped at a pressure of 750-1500 psi and a rate of 2 gal/min through a two fluid nozzle, with steam as the heating medium at a pressure of 140-170 psig achieving cooking chamber temperatures of 300°-400°F and thereby gelatinizing or cooking the starch granules. This product is then dried by passing the cooked slurry stream through a spray drying tower. By processing as indicated, certain modified potato starches are 15 cooked to produce whole granular pregelatinized starches (cold water swelling starches) which behave contrary to the expected behavior of viscosity reduction with regards to processing stress such as shear. These starches substantially gain in viscosity following moderate (*e.g.*, Hobart™ mixer) to high shear (*e.g.*, Waring™ blender, Silverson™ Rotostator 20 Mixer, colloid mill) of the kinds usually employed in the food, cosmetics and personal care industries, to list a few. Other modified/crosslinked, pregelatinized non-cereal starches having similar properties (see Figure 1 and its description) would be suitable according to the invention. Also, other methods of producing whole granular pregelatinized starches such as, but not limited to, those detailed in U.S. Patent No. 3,617,383 and U.S. Patent No. 5,057,157 25 or derivative patents thereto are included in this preferred embodiment.

In the preferred embodiment, the invention provides for edible pregelatinized modified potato starches produced by atomized cooking which have been derivatized by standard methods by treatment with propylene oxide to produce a degree of substitution (DS) 30 in the range of up to 0.22 and a preferred range of 0.8-0.16. Further it provides for pregelatinized starches which have been crosslinked by standard procedures with an amount of phosphorous oxychloride in the range of 75-1000ppm and a preferred range of 75-400ppm

and/or substituted as per the above conditions. While the preferred starch is the Granular CWS high cross-linked potato starch in Table 1 as described above, other so crosslinked/modified non-cereal starches would also be suitable to the invention. The data in Example 1 and Table 1 detail and exemplify these findings.

5

In the most preferred embodiment of this invention the rheological profile for the starch thus produced, the preferred starch, can be described as a manifestation of "thin-thick" behavior. This occurs under basic pH (>7.0), at neutrality (pH=7.0) and under acid conditions. The magnitude of the viscosity increase with high shear ("thin-thick" behavior) is not observed for the same modified potato source starch when pregelatinized by other methods which yield a starch with a minority of intact granules (e.g., roll drum dried), for other lesser crosslinked potato starches (Figure 1) or for similarly modified/ crosslinked starches from cereal raw material sources (e.g., waxy maize, corn). The data in Example 1 and Tables 2-4 exemplify and support these findings in a model food system.

10

These are unique properties for a pregelatinized starch which have not been reported previously and which may have useful applications in numerous use areas (eg., food processing, cosmetics, personal care, etc.). The food applications where this "thin-thick" rheological profile would be useful can include, but are not limited to, pourable salad dressings, spoonable mayonnaise or dressings and fruit preparations, toothpaste. The reduction to practice of the preferred embodiment starch in a pourable salad dressing application is outlined in Example 2 and Table 5 and exemplified in Figure 3. A practical application in a practical fruit preparation is detailed in Example 3 and Table 6.

15

The pregelatinized starches of the invention are relatively stable in terms of freeze/thaw, refrigerated storage, heat and shear, yet can be easily further processed at low, workable viscosities. These starches build higher, desirable product viscosities upon application of significant mechanical action, e.g., shear by various mixers, Lightnin™ Mixer, Waring™ blender, colloid mill, etc. Such characteristics are advantageous and desirable in a thickening or rheological ingredient for products of several industries including food processing, cosmetics, personal care, and others. Food applications where this thin-to-thick ("thin-thick") rheological profile would be useful include, but are not limited to, pourable

20

25

30

salad dressings, spoonable mayonnaise and dressings and fruit preparations. Cosmetic and personal care applications include, but are not limited to, skin and body lotions, hair creams and preparations and cosmetic creams and lotions.

5 Yet another preferred embodiment of this invention involves the use of pregelatinized starches modified and processed as noted above (the preferred starch) as a replacement for costly hydrocolloids such as xanthan gum for reasons of suspension, viscosity and clarity. They would find utility in food applications such as salad dressings (e.g., fat free Italian salad dressing) where small particles of spices and seasonings are required to become and remain suspended over time in a relatively clear liquid product. It is highly advantageous for the liquid fraction to remain at a low, workable viscosity until shearing with equipment such as a colloid mill after which it is required to develop and maintain sufficient viscosity to suspend the spice and seasoning over substantial time. As outlined in Example 4 and supported by data in Table 7, the findings in this regard of this invention are exemplified.

10  
15 Another preferred embodiment of this invention involves the use of these pregelatinized starches modified and processed as noted above (the preferred starch) as a viscofying hydrocolloid in cosmetic and personal care preparations. In such products as shown in Example 5, the starch acts to bind water, provide sufficient viscosity to suspend solids  
20 while imparting a smooth sensory feel to the preparations. The starches impart the clear advantage of allowing all necessary mixing, pumping and emulsion manipulation to occur at relatively low working viscosities prior to application of sufficient shear to induce their shear thickening properties and impart the final higher viscosity and stability to the cosmetic or personal care preparations. As per the formulation cited in Example 5 which contains an alpha  
25 hydroxy acid, another benefit of the use of the preferred starch is its shear thickening and stable viscosity under acid conditions (pH <4.0).

30 The following Example is are provided to further illustrate and explain a preferred form of the invention and are not to be taken as limiting in any regard. Unless otherwise indicated, all parts and percentages are by weight.

**Example 1**

This example describes the rheological properties of the starches, such as the preferred starch, which is the subject of the preferred embodiment of this invention. This rheology can 5 be described as "thin-thick" behavior and is characterized more precisely in Figure 1 and 2 in terms of the viscosity of the model food system of Example 1. This thin-thick behavior occurs both at neutrality (pH=7.0) and under acid conditions. The large magnitude viscosity increase with high shear is not seen for the same source modified potato starch when cooked by methods producing non-granular pregelatinized starch (e.g., roll drum dried), for potato 10 starches crosslinked at lesser levels or for similarly modified/crosslinked starches from cereal sources (e.g. , waxy maize, corn). Figures 1 and 2 and the data in Tables 2-4 exemplify and support these findings in a model food system.

As a first stage in the process, a Granular CWS high cross-linked potato starch as 15 defined above in Table 1 is subjected to heating practicing the atomized cooking technology detailed in U.S. Patent Nos. 4,280,851, 4,600,472 and 4,847,371. Specifically, a 30-40% solids starch slurry at a pH = 6.0 – 7.5 is pumped at a pressure of 750-1500 psi and a rate of 2 gal/min through a two fluid nozzle, with steam as the heating medium at a pressure of 140- 20 170 psig achieving cooking chamber temperatures of 300°- 400°F and thereby gelatinizing or cooking the starch granules. This product is then dried by passing the cooked slurry stream through a spray drying tower. The product has substantially all of the starch granules intact and is cold water swelling.

For the purposes of our test, the following model food system was prepared and 25 processed according to the following procedure.

**Model Food System**

	<b>Ingredient</b>	<b>%</b>
30	Sugar	23.50
	Water	40.00
	Vinegar	30.00
	Salt	2.50
35	<b>Starch</b>	<b>4.00</b>
	Total	100.0

**Procedure**

1. The sugar, salt, and starch were dry blended together.
2. The dry ingredients were added to water using low shear overhead mixer.
3. The slurry was then subjected to high shear using a Waring blender or Silverson Rotostator Mixer as noted.

5

**Table 2. Pregelatinized Starch Viscosity vs. Time and Shear  
(Pregelatinization by Spray Cooking, Slurry pH=3.0)**

10

Starch	1 min. (cps) Low Shear*	20min. (cps) Low Shear*	24hr. (cps) Low Shear*	24hr. (cps) w/High Shear <sup>#</sup>
Granular CWS medium cross-linked waxy maize starch	300	810	3900	1500
Granular CWS high cross-linked waxy maize starch	550	1000	4000	1100
Granular CWS medium cross-linked potato starch	2160	2180	6080	720
Granular CWS high cross-linked potato starch	380	450	680	2000

\*Low Shear = Overhead mixer

<sup>#</sup>High Shear = Waring blender, 1 min. after 24hr. Low Shear

All Viscosities: Brookfield viscometer, Spindle RVT4, RPM=10

15

 100  
90  
80  
70  
60  
50  
40  
30  
20  
10  
0

**Table 3. Pregelatinized Starch Viscosity vs. Granularity, Time, Shear and pH**

Starch	pH	Low Shear* 1min. (cps)	Low Shear* 5min. (cps)	High Shear# 1min. (cps)
Non-Granular CWS high cross-linked potato starch	3.0	910	4620	2300
Preferred Starch	3.0	460	600	4600
Non-Granular CWS high cross-linked potato starch	7.0	2500	5180	2900
Granular CWS high cross-linked potato starch	7.0	960	1000	5200

\*Low Shear = Overhead Mixer

#High Shear = Silverson Rotostator Mixer, 30 sec. @ 8400rpm after 5min. Low Shear

All Viscosities: Brookfield viscometer, Spindle RVT4, RPM=10

**Table 4. Pregelatinized Starch Viscosity vs. Degree of Crosslinking, Time and Shear (Pregelatinization by Spray Cooking, Slurry pH = 3.0.)**

Starch	Low Shear* 1min. (cps)	Low Shear* 5min. (cps)	High Shear# 1min. (cps)
Granular CWS low cross-linked potato starch	2020	5300	320
Granular CWS medium cross-linked potato starch	2600	8300	2100
Granular CWS high cross-linked potato starch	460	600	4600

\*Low Shear = Overhead Mixer

#High Shear = Silverson Rotostator Mixer, 30 sec. @ 8400rpm after 5min. Low Shear

All Viscosities: Brookfield viscometer, Spindle RVT4, RPM=10

**Example 2**

This example describes a practical application of the preferred embodiment of this invention. It is highly advantageous that such starches be stable in high shear processing since 5 traditional manufacture includes passing the product through a colloid mill, homogenizer or similar high shear equipment to produce the final dressing. It is also very advantageous for manufacturers to be able to carry out all the mixing, blending, pumping and salad dressing formulating procedures at low working viscosities prior to passing it through the colloid mill to produce the bottled product with its final higher viscosity. Starches such as the preferred 10 starch fit the needs of this application extremely well owing to the "thin-thick" rheological behavior it displays as exemplified in Example 1 above.

15 Fat free French salad dressings were prepared following the formulation and procedure listed below using the preferred starch, comparably modified waxy maize, and a comparably modified cornstarch. The shearing of the salad dressings was carried out on an APV model 2F colloid mill using a 3 pass procedure through a .005"gap. Brookfield viscosities were measured after the low-shear mixing step, after colloid mill shearing and again in 20 minutes as indicated in Table 5. It can be seen that there was a greater than 11-fold increase in viscosity observed with the preferred starch which was dramatically greater 20 than the viscosities developed by the dressings with either of the cereal starches.

**Fat Free French Salad Dressing**

	<b>Ingredients</b>	<b>%</b>
25	Water	33.66
	High Fructose Corn Syrup	39.54
	Tomato Sauce (9.5% Solids)	12.00
	Vinegar (120 Grain)	8.00
	Starch	3.00
30	Salt	2.50
	Onion Powder	0.85
	Titanium Dioxide	0.07
	Xanthan	0.15
	Potassium Sorbate	0.09
35	Sodium Benzoate	0.09
	EDTA	0.005
	<u>Yellow Color #6</u>	<u>0.04</u>
	Total	100.00

**Procedure:**

1. Add Water to blender or tank.
2. Add Sodium Benzoate and Potassium Sorbate and mix at low speed.
- 5 3. Add Vinegar and High Fructose Corn Syrup.
4. Mix together all dry ingredients.
5. Add premixed dry ingredients to blender or tank and mix briefly at low speed. The viscosity should be low at this point.
6. Process through a colloid mill. Note the dramatic viscosity increase.
- 10 7. Add tomato sauce and blend briefly at medium speed.

**Table 5. Fat Free French Dressing Viscosities**

Starch	Viscosity (cps) Low Shear *	Viscosity (cps) High Shear#	Viscosity (cps) High Shear+20min.
Preferred starch	600	5660	6900
Granular CWS high cross-linked waxy maize starch	700	2820	3240
Granular CWS high cross-linked corn starch	640	2320	2840

15 \*Low Shear = Overhead Mixer

# High Shear = APV 2F Colloid Mill, .005"gap/3-pass

All Viscosities: Brookfield viscometer, Spindle RVT4, RPM=10

20

**Example 3**

This example describes another reduction to practice of the preferred embodiment of this invention. In systems with minimal water available such as fruit preparations starches have been used to assist gelling by providing viscosity and to impart soft texture. The 25 commercial processes for manufacture of these preparations are typically quite severe involving temperatures of 200° F or more, holding times of 30 minutes or greater, pH in the range of 3-4 and moderate/high shear. It is highly advantageous for producers to be able to carry out this processing at lower viscosities without sacrificing loss of final product viscosity due to partial starch breakdown. Starches such as the preferred starch are very well suited to 30 this need owing to their rheological properties as have been demonstrated in Example 1 above.

Fruit preparations were made following the formulation and procedure listed below. Using the preferred modified potato starch, and a comparably modified waxy maize starch. Heating to 200° F was carried out for 2 minutes followed by cooling to 100°F. The preparations were then blended using low shear overhead mixing followed by high shear 5 milling with a Silverson mixer. They had pH values of 3.4. Brookfield viscosities were taken after the low shear mixing and again after the high shear treatment as shown in Table 6. The fruit preparation with the preferred starch had a viscosity after low shear mixing of 60cps vs. 200cps for comparably modified waxy maize. Within one hour after high shear, the fruit preparation with the preferred starch had a viscosity of 2100cps which is 35 times greater 10 than after low shear mixing and 6-7 times that of the fruit preparation with the comparably modified waxy maize starch. The preferred starch clearly demonstrated its propensity for shear thickening in this application and its overwhelming functional superiority to the comparably modified waxy maize starch in this regard.

#### **Cranberry Fruit Preparation**

Ingredients	%
<b>70 Brix Syrup</b>	
Cranberry Juice	66.6
Sucrose	<u>33.4</u>
	100.0
<b>Model System</b>	
70 Brix Syrup	48.50
Water	48.50
<u>Starch</u>	<u>3.00</u>
Total	100.00

#### **Procedure**

1. Combine cranberry juice and sucrose in a saucepan.
2. Heat to 200° F stirring continually until sucrose is completely dissolved.
3. Allow solution to cool to 100° F.
4. Combine the correct amount of 70 brix solution with water and starch and stir using a propellar mixer at 320 rpm for 1 minute. Measure viscosity.
5. Shear slurry on a Silverson mixer at 9000 rpm for 10 minutes (square stator).
6. Measure viscosity.

Table 6. Fruit Preparation Viscosities (Preparation pH = 3.4)

Starch	Low Shear*	High Shear#	Shear + 20 min.	Shear + 1 hr.
	Viscosity (cps)	Viscosity (cps)	Viscosity (cps)	Viscosity (cps)
Preferred Starch	60	560	1680	2100
Granular CWS high cross-linked waxy maize starch	200	200	260	380

All Viscosities: Brookfield viscometer, Spindle RTV4, RPM=10

5 \* Low Shear = Overhead Mixer, 320rpm, 1 min.

# High Shear = Siverson Rotostator Mixer, 30 sec. @ 9000rpm after Low Shear

#### Example 4

This example describes another practical food application of the preferred embodiment of this invention. Xanthan gum, a relatively expensive hydrocolloid, is used widely in the preparation of salad dressings where particulate suspension, viscosity and clarity are required. It is especially useful in that it provides high viscosity even after shearing which is functionally desirable in salad dressings in which small particles of spice and seasonings need to be suspended over time in a clear liquid (e.g., fat free Italian dressing). At a typical 0.6% usage level, this allows for low working viscosities for processing ease while developing sufficient final product viscosity after shearing to allow for particulate suspension in the bottled dressing. The preferred starch provides many of the key functionalities of xanthan gum

20 in that modified potato starch provides a clear slurry and the preferred starch, a cold water swelling highly crosslinked potato starch, also imparts high viscosity after shearing. The preferred starch seems to be a well-suited, cost-effective replacement for xanthan gum in such applications.

25 Salad dressing was prepared following the formulation and procedure listed below. The preferred starch was used at 2.7 % as the starch component and the dressing so prepared was compared to one similarly produced containing 0.6% xanthan gum. Both formulations were fluid with low, workable viscosities in the mixing stages. Brookfield viscosities, clarity and particulate suspension were compared one week after preparation and bottling. It can be

seen from the data in Table 7 that the dressing made with the preferred starch had a one week viscosity of 2400cps, exhibited excellent clarity and was able to keep the spice and seasoning particles suspended with no settling noted. In these respects, it was equivalent to the dressing containing the xanthan gum. As such, the preferred starch has been shown to be an effective  
 5 shear thickening, hydrocolloid replacement for xanthan gum in such applications. The cost and use benefits of such a replacement would be apparent to those skilled in salad dressing formulation.

#### Fat Free Italian Dressing

10	Ingredients	%
15	Water	17.75
20	Preferred Starch	2.70
25	HFCS 42 %	27.22
30	Vinegar 50 Grain	45.74
35	Salt	4.54
40	Onion - Minced	0.72
45	Garlic - Minced	0.54
50	Black Pepper - Coarse Grind	0.25
55	Monterey Seasoning Blend	0.36
60	<u>Dehydrated Vegetable Blend</u>	<u>0.18</u>
65	Total	100.00

#### Procedure

1. Add water to blender or batch tank.
2. Add sodium benzoate and potassium sorbate and mix briefly at low speed.
3. Add vinegar and high fructose corn syrup(HFCS 42%).
4. Mix together all dry ingredient except spices.
5. Add premixed dry ingredients and mix briefly at low speed. Observe the viscosity.
6. Blend at highest speed for 30 seconds or process in colloid mill. Note dramatic viscosity increase.
7. Add all spices and blend or mix briefly at medium speed.

35 **Table 7. Fat Free Italian Dressing Viscosities and Functionalities**

Hydrocolloid	Usage(%)	Viscosity(cps) 1 week*	Clarity	Particulate Suspension
Xanthan Gum	0.6	3900	Excellent	No Settling
Preferred Starch	2.7	2400	Excellent	No Settling

\* After High Shear: APV 2F Colloid Mill  
 All Viscosities: Brookfield viscometer, Spindle RVT4, RPM=10

**Example 5**

This example describes a practical application of the preferred embodiment of this invention in the field of cosmetics and personal care products. Cosmetic and personal care lotions and creams for the skin, body and hair often employ starches as viscosifying agents and hydrocolloids. The starch functions as a water binder and imparts viscosity sufficient to suspend solids while adding to the smooth feel of the preparations. It has been found that the preferred starch imparts all these functional benefits to cosmetic preparations while allowing all processing mixing, pumping and emulsion manipulation to occur with ease at low working viscosities due to its thin-thick rheological properties as demonstrated in Example 1. With application of high shear just prior to completion of product manufacture the shear thickening properties are induced and the higher final product viscosities and stability desired are achieved in the cosmetic or personal care preparation. Moreover, as was demonstrated in Example 1, another benefit of the use of the preferred starch is its shear thickening and stable viscosity under acidic conditions. This makes it especially well-suited for use in acidic cosmetic lotions (e.g., skin lotions with alpha hydroxyacid).

Using the formulation and following the procedure below, a skin lotion preparation containing alpha hydroxyacid was made. The preferred starch was used at the 2% level as part of Phase A of the lotion formulation. Phase A containing the starch was subjected to low shear with an overhead mixer and heated to 120°F to uniformly blend all ingredients. Phase B was formed by melting the waxy components at 140°F. It was added to Phase A while low speed homogenizing. The liquid Phase C was added to the AB homogenate mixture and the entire formulation (ABC) was subjected to 10 minutes of high speed homogenization to produce the final lotion (pH=3.4).

Viscosities were measured after forming the ABC Phase composite and again after the 10 minute high speed homogenization. It can be seen in Table 8 that the lotion viscosity following high shear homogenization increased nearly 50% over the ABC composite (4200cps vs 2800cps) which had already been subjected to a combined moderate shear. This is another clear manifestation of the shear thickening properties of the preferred starch. The advantages of using a starch with the demonstrated rheological behavior of the preferred starch, especially

in an acidic cosmetic lotion, are obvious to those skilled in the formulation of cosmetic and personal care preparations.

5 **Skin Lotion with Alpha Hydroxy Acid**

Phase	Ingredient	%
A	Water	59.4
A	Methyl Paraben	0.3
A	Germall 2	0.3
A	Preferred Starch	2.0
B	Isopropyl Palmitate	20.0
B	Cetyl Alcohol	1.0
B	Glyceryl Monostearate/PEG-100 Stearate	2.0
C	Glycolic Acid	5.0
C	Water	10.0
		100.0

20 **Procedure**

1. Disperse sequentially the Phase A dry ingredient in the water using an overhead mixer.
2. Heat all Phase B ingredients to 140°F until waxy components fully melt together.
3. Heat Phase A to 120°F.
4. Add Phase B(melted) to Phase A while homogenizing on slow speed. Air cool to 95°F.
5. Dissolve Phase C glycolic acid and add to AB.
6. Homogenize entire formulation on high for 10 minutes.

30 **Table 8. Viscosity of Skin Lotion with Alpha Hydroxy Acid (Lotion pH =3.4)**

Starch	Viscosity(cps) Low/Moderate Shear*	Viscosity(cps) High Shear #
Preferred Starch	2800	4200

\* Low/Moderate Shear: Overhead Mixer; Gifford-Wood Homogenizer, low speed

# High Shear: Gifford-Wood Homogenizer, high speed/10 minutes

35 All Viscosities: Brookfield viscometer, Spindle RTV 4, RPM=20

The above description is intended to enable the person skilled in the art to practice the invention. It is not intended to detail all of the possible modifications and variations

which will become apparent to the skilled worker upon reading the description. It is intended, however, that all such modifications and variations be included within the scope of the invention which is seen in the above description and otherwise defined by the following claims. The claims are meant to cover the indicated elements and steps in any arrangement or sequence which is effective to meet the objectives intended for the invention, unless the context specifically indicates the contrary.

5 or sequence which is effec

## CLAIMS

1. A process for preparing pregelatinized, cold water swelling starch, comprising: providing a highly cross linked, stabilized starch; and heating said starch under conditions of time, temperature and moisture effective to disrupt less than 50% of intact starch granules comprised in said starch and to provide the characteristic that when a slurry of said starch is subjected to high shear the viscosity will increase toward a maximum and retain that viscosity throughout further low shear mixing and holding periods.
2. A process according to Claim 1 wherein said starch is a cross-linked by treatment with phosphorous oxychloride or sodium trimetaphosphate.
3. A process according to Claim 1 or 2 wherein said starch is a stabilized starch, such as an hydroxypropylated, acetylated, phosphorylated, dextrinized starch or a starch substituted with octenylsuccinate groups.
4. A process according to Claim 1, 2 or 3 wherein at least a majority of the starch granules are whole and unbroken.
5. A process according to Claim 1, 2, 3 or 4 wherein said starch is a potato starch.
6. A process according to Claim 1, 2, 3 or 4 wherein said starch is a tapioca starch.
7. A process according to Claim 1, 2, 3 or 4 wherein said starch is derived from any one or combination of the following: potato starch, tapioca starch, arrowroot starch, yam starch, sweet potato starch.
8. A process according to Claim 1, 2, 3 or 4 wherein said starch is a non-cereal starch.

9. A pregelatinized starch product of non-cereal origin which exhibits notable shear thickening properties following medium to high shear mixing thereby enhancing their viscometric properties.
10. A pregelatinized starch product, which exhibits shear-thickening properties, comprising: a highly crosslinked, stabilized and cold water swelling starch having a majority of its starch granules intact and being characterized in that when a slurry of said starch is subjected to high shear the viscosity will increase toward a maximum and retain that viscosity through out further low shear mixing and holding periods.
11. A pregelatinized starch product according to claim 10 which exhibits an increase in viscosity to a maximum following high shear mixing of an aqueous slurry wherein the final viscosity following mixing and holding to form a product is no less than 25% of the maximum viscosity.
12. A pregelatinized starch product according to claim 10 which is characterized in that an aqueous slurry does not significantly increase viscosity under low shear conditions, the increase being less than 50% of the maximum achievable under high shear conditions.
13. A pregelatinized starch product according to claim 10 which is characterized in an aqueous slurry exhibiting not less than 50% of the viscosity values of the preferred starch illustrated in Figure 1 following high shear mixing.
14. A pregelatinized starch product according to claim 10 which is characterized in an aqueous slurry exhibiting a viscosity after high shear at least 2 times as high as that exhibited after low shear and should not decrease in viscosity from a maximum by more than about 25%.
15. A pregelatinized starch product according to claim 10 which is characterized in an aqueous slurry exhibiting a viscosity after high shear at least 6 times as high as

that exhibited after low shear and should not decrease in viscosity from a maximum by more than about 25%.

16. A foodstuff containing a pregelatinized starch as in Claim 9 wherein the foodstuff has a pH of 2.5 to 8.0.
17. A cosmetic or personal care product containing a pregelatinized starch as in Claim 10, 11, 12, 13, 14 or 15 wherein the cosmetic or personal care product has a pH of greater than 8.0.
18. A pregelatinized starch as in Claim 10, 11, 12, 13, 14 or 15 wherein said starch hydrates at a sufficiently slow rate as to be easily dispersible.
19. A pregelatinized starch as in Claim 10, 11, 12, 13, 14 or 15 wherein said starch is a potato starch or arrowroot starch with granular size greater than 25 microns.
20. A pregelatinized starch as in Claim 10, 11, 12, 13, 14 or 15 that can be used in place of other hydrocolloids such as xanthan gum for reasons of suspension, viscosity, clarity.
21. A pregelatinized starch as in Claim 10, 11, 12, 13, 14 or 15 that allows for reduction in use level over cereal starches in foodstuffs such as pourable salad dressing of 5-30%.
22. A pregelatinized starch for use in foodstuff, cosmetic or personal care products for use according to any one of Claims 10, 11, 12, 13, 14 or 15.
23. A foodstuff obtainable using a pregelatinized starch such as those in any one of Claims 10, 11, 12, 13, 14 or 15.
24. A cosmetic or personal care product using a pregelatinized starch such as those in any one of the Claims 10, 11, 12, 13, 14 or 15.

25. Pregelatinized starch products of non-cereal origin and compositions which exhibit notable shear thickening properties following processing stress thereby enhancing their viscometric properties and utilities in foodstuff, cosmetics and personal care products.

TRIESTE CENTER

Figure 1.

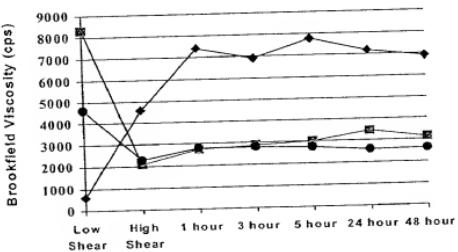


Figure 2.

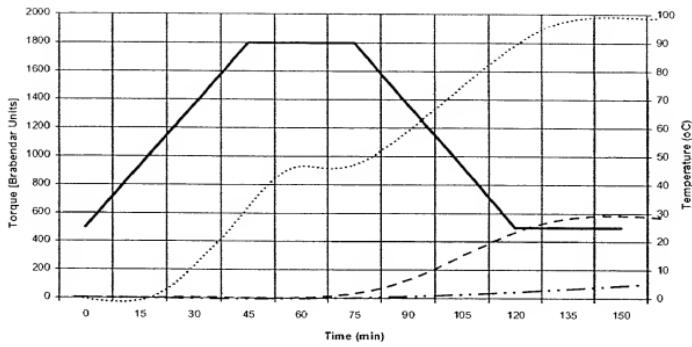
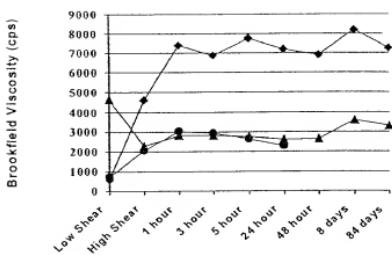
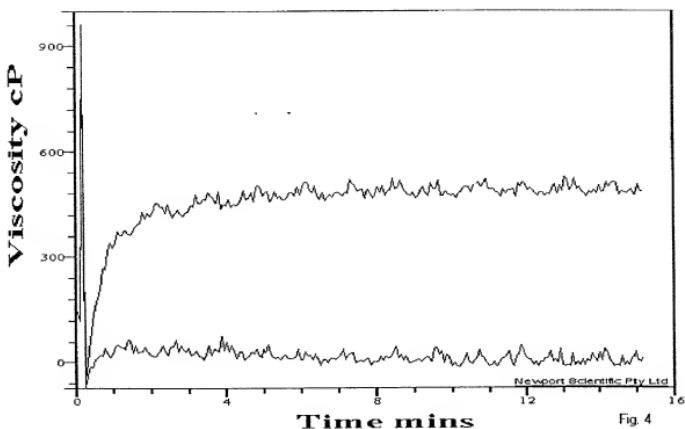


Figure 3.



**Figure 4**



PATENT  
835-013-011-2

Declaration and Power of Attorney

As below-named inventors, we hereby declare that:

Our residences, post office addresses, and citizenships are as stated below next to our names.

We believe that we are the original, first and joint inventors of the subject matter which is claimed and for which a patent is sought on the invention entitled **Shear Thickening Pregelatinized Starch**, bearing Serial No 09/831,836, filed May 15, 2001. We hereby state that we have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above. To the best of our knowledge, information, and belief the facts stated therein are true.

We acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

We hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign applications for patent or inventor's certificate listed below and have also identified below any foreign applications for patent or inventor's certificate having a filing date before that of the application on which priority is claimed.

None

We hereby claim the benefit under Title 35, United States Code, §120 of the following United States Applications listed below, and insofar as the subject matter of each of the claims of this Application is not disclosed in the prior United States Application in the manner provided by the first paragraph of Title 35, United States Code, §112, we acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, §1.56 which occurred between the filing date of the prior application and the national or PCT international filing date of this application.

U.S. Provisional Application Serial No. Serial 60/154,081, Filed September 15, 1999.

We hereby declare that all statements made herein of our own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

✓ We hereby appoint Thaddius J. Carvis, Registration No. 26,110, of Ware, Fressola, Van Der Sluys & Adolphson LLP, Bradford Green, Building Five, 755 Main Street, P.O. Box 224, Monroe, CT 06468, (203) 261-1234 with full power of substitution, association and revocation, as attorney to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith.

Please direct all telephone calls and correspondence to Thaddius J. Carvis at the above address and telephone number.

Full name of first inventor:

Charles J. Brine

Charles J. Brine

JULY 30, 2001

NJ

Princeton, New Jersey 08540

Inventor's signature

Date:

Residence:

Citizenship:

Post Office Address:

A citizen of United States of America

28 Tee-Ar Place

Princeton, NJ 08540

Full name of second inventor:

Anne E. Tielemans

Anne E. Tielemans

Inventor's signature

Date:

JULY 30, 2001

PA

Langhorne, Pennsylvania 19074

Residence:

Citizenship:

A citizen of United States of America

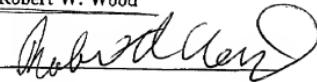
Post Office Address:

45 Gunning Lane

Langhorne, PA 19074

300  
Full name of third inventor:

Robert W. Wood



Inventor's signature

Date:

JULY 31 2001

NJ

Residence:

Cranbury, New Jersey 08512

Citizenship:

A citizen of United States of America

Post Office Address:

5 Wynnewood Drive  
Cranbury, NJ 08512

United States Patent & Trademark Office  
Office of Initial Patent Examination – Scanning Division



Application deficiencies found during scanning:

Page(s) 2-9 of Transmittal letter were not present  
for scanning. (Document title)

Page(s) \_\_\_\_\_ of \_\_\_\_\_ were not present  
for scanning. (Document title)

*Scanned copy is best available.*